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PHOTOREACTION OF HEXACARBONYLMOLYBDENUM(O) AND TRICARBONYL(ETA(5)-
JUL 79 W C MILLS, M S WRIGHTON N00014-75-C-0880

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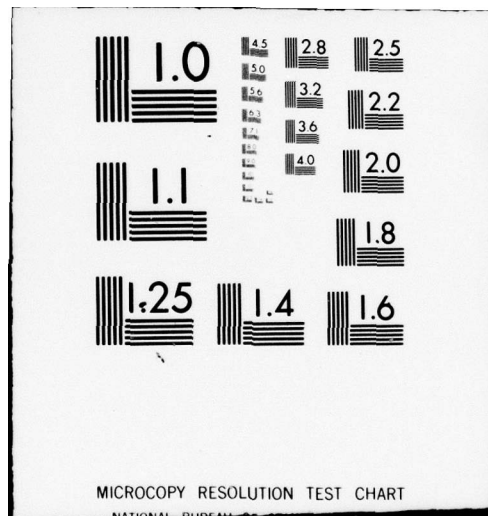
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6 Photoreaction of Hexacarbonylmolybdenum(0) and
Tricarbonyl(η^5 -cyclopentadienyl)hydridomolybdenum(II) in the
Presence of Cyclopentadiene: Substitution, Oxidative Addition,
and Hydrometallation

by

10 William C. Mills, III and Mark S. Wrighton

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trans-trans-2,4-hexadiene, 1,4-pentadiene, trans-1,3-pentadiene also appears to obtain upon irradiation of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3\text{H}$ in the presence of the diene. Irradiation of $\text{Cr}(\text{CO})_6$ in the presence of C_5H_6 yields $\text{Cr}(\text{CO})_2(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-C}_5\text{H}_7)$.

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Photoreaction of Hexacarbonylmolybdenum(0) and Tricarbonyl(η^5 -cyclopentadienyl)hydridomolybdenum(II) in the Presence of Cyclopentadiene: Substitution, Oxidative Addition, and Hydrometallation

Abstract: Irradiation of Mo(CO)_6 in the presence of C_5H_6 yields $(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)\text{Mo(CO)}_2$ via a sequence of light-induced reactions involving substitution, $\text{Mo(CO)}_n(\text{C}_5\text{H}_6)$ ($n = 5, 4$); oxidative addition, $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$; and hydrometallation, $(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)\text{Mo(CO)}_2$. The sequence of events can be followed by monitoring the infrared spectral changes in the CO stretching region. An authentic sample of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ cleanly yields $(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)\text{Mo(CO)}_2$ when irradiated in the presence of C_5H_6 ; the 355 nm quantum yield for this reaction is 0.12 ± 0.02 . Photoinduced hydrometallation of $\text{C}_5(\text{CH}_3)_5\text{H}$, trans-trans-2,4-hexadiene, 1,4-pentadiene, trans-1,3-pentadiene also appears to obtain upon irradiation of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ in the presence of the diene. Irradiation of Cr(CO)_6 in the presence of C_5H_6 yields $\text{Cr(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$.

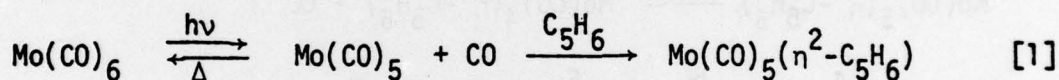
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Sir:

We wish to report new results pertaining to photoreaction of low-valent metal carbonyls in the presence of olefins. The systems reported on here are Mo(CO)_6 and $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ irradiated in the presence of cyclopentadiene, C_5H_6 . These particular systems are important inasmuch as Mo(CO)_6 is a known catalyst precursor under photochemical conditions for the isomerization of olefins¹ and 1,4-hydrogenation of 1,3-dienes,² and $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ is reported to be a stoichiometric reducing agent for converting 1,3-dienes to alkenes.³ Further, C_5H_6 is a source of $\eta^5\text{-C}_5\text{H}_5$ in metal complexes, when reacted with an appropriate precursor. Generally, using light to effect an individual step in a catalytic or stoichiometric process may provide for greater specificity or rate or change the course of events altogether. In the systems under consideration here, Mo-CO dissociation is likely a thermally rate limiting process at room temperature which can be substantively altered by optical excitation.^{4,5}

Our first interest in the $\text{Mo(CO)}_6/\text{C}_5\text{H}_6$ system actually began some years ago when we attempted the photoassisted 1,4-hydrogenation of C_5H_6 to cyclopentene under the conditions² giving efficient hydrogenation of those 1,3-dienes which can easily achieve, or are held in, an s-cis conformation. Little catalytic chemistry occurred using Cr(CO)_6 the catalyst precursor in the attempted hydrogenation of C_5H_6 , though large

spectral changes obtained upon irradiation. We now present the results of a study of the irradiation of Mo(CO)_6 or Cr(CO)_6 in the presence of C_5H_6 . Irradiation (355 ± 20 nm, 2×10^{-6} ein/min) at 25°C of $5 \times 10^{-3}\text{M}$ Mo(CO)_6 in a degassed isooctane solution of freshly distilled 0.1M C_5H_6 rapidly yields chemical reaction which can be monitored by ir spectroscopy in the CO-stretching region. In accord with very early findings associated with irradiation of M(CO)_6 ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) in the presence of olefins,⁶ the ir spectral changes initially correspond to formation of $\text{Mo(CO)}_5(\eta^2\text{-C}_5\text{H}_6)$ with ir bands as given in Table I, equation (1). This primary photoproduct, however, absorbs in the near-uv

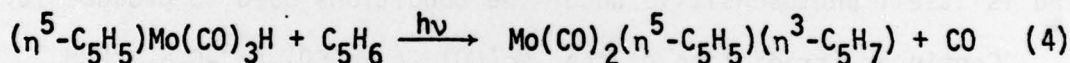
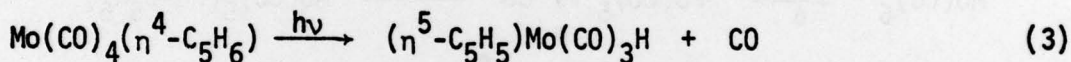
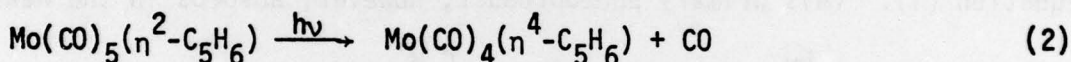


and is itself photosensitive under the conditions used to produce it.

Continued irradiation of the $\text{Mo(CO)}_6 / \text{Mo(CO)}_5(\eta^2\text{-C}_5\text{H}_6) / \text{C}_5\text{H}_6$ mixture results in the further decline of Mo(CO)_6 and growth of other Mo-carbonyl species. Ir bands of greater and lesser importance appear at 2043(w), 2029(w), 1961(s), and 1912(w), cm^{-1} and ultimately, two prominent features at 1880 and 1950 cm^{-1} appear. The strong 1961 cm^{-1} feature is attributable to trans- $\text{Mo(CO)}_4(\eta^2\text{-C}_5\text{H}_6)_2$ on the basis of its intensity dependence in time and the fact that trans- $\text{Mo(CO)}_4(\eta^{2-1,3}\text{-butadiene})_2$ has a similar feature at 1965 cm^{-1} .⁶ The bands at 2043 and 1912 cm^{-1} may be due to $\text{Mo(CO)}_4(\eta^4\text{-C}_5\text{H}_6)$ or to cis- $\text{Mo(CO)}_4(\eta^2\text{-C}_5\text{H}_6)_2$; presumably the other two features for such complexes⁷ in the 1965-1940 cm^{-1} range are obscured by the 1961 and 1957 cm^{-1} absorptions of trans- $\text{Mo(CO)}_4(\eta^2\text{-C}_5\text{H}_6)_2$ and $\text{Mo(CO)}_5(\eta^2\text{-C}_5\text{H}_6)$, respectively. The weak band at 2029 cm^{-1} identically matches the highest energy CO-stretching absorption of an authentic sample of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ ⁸, but

the 1946 cm^{-1} band of this complex is obscured. The bands at 1950 and 1880 cm^{-1} identically match those from an authentic sample of $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$.⁹ This product is somewhat photosensitive, and it, too, ultimately reacts. Yields of up to ~20% (based on ir absorption) of $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$ can be realized starting with Mo(CO)_6 . Interestingly, the irradiation of Cr(CO)_6 in the presence of C_5H_6 results in ir bands at 1942 and 1880 cm^{-1} which we attribute to $\text{Cr(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$.¹⁰ The formation of such products possibly accounts for our inability to effect the 1,4-hydrogenation of C_5H_6 at 1 atm. H_2 pressure.

Equations (1)-(4) represent a plausible sequence of photochemical



reactions leading to the formation of the $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$. Irradiation of trans- $\text{Mo(CO)}_4(\eta^2\text{-C}_5\text{H}_6)_2$ may also give rise to this product; isolation and characterization of both tetracarbonyl species is of interest. But whatever the mechanism, the product reflects the fact that C_5H_6 binds, oxidatively adds, and undergoes hydrometallation. The reactive, doubly allylic C-H bonds and the stability of $\eta^5\text{-C}_5\text{H}_5$ complexes promotes the formation of the $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ upon photogeneration of coordinatively unsaturated intermediates in the presence of C_5H_6 .

Irradiation of an authentic sample of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ ¹¹ in the presence of C_5H_6 leads to the clean formation of $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$ ¹² according to equation (4). Inasmuch as the hydride is conveniently synthesized, equation (4) represents the synthetic procedure of choice for $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$. The 355 nm quantum yield is 0.12 ± 0.02 . Figure 1 shows the ir spectral changes accompanying a typical photoreaction. We conclude that the formation of product proceeds via photogeneration of $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_2\text{H}$ followed by thermal

reaction with C_5H_6 . An alternative mechanism involving $(\eta^5-C_5H_5)Mo(CO)_3$ radicals as in the simple substitution of $(\eta^5-C_5H_5)Mo(CO)_3H$ ¹³ appears to be ruled out by the following experiment. Visible light (514.5 nm) irradiation of $(\eta^5-C_5H_5)_2Mo_2(CO)_6$ in the presence of $(\eta^5-C_5H_5)Mo(CO)_3H$ and C_5H_6 does not lead to $Mo(CO)_2(\eta^5-C_5H_5)(\eta^3-C_5H_7)$. $(\eta^5-C_5H_5)_2Mo_2(CO)_6$ is a known photochemical source of $(\eta^5-C_5H_5)Mo(CO)_3$ radicals.¹⁴ Irradiation of $(\eta^5-C_5H_5)Mo(CO)_3H$ in the presence of other dienes such as trans-1,3-pentadiene, trans,trans-2,4-hexadiene, 1,4-pentadiene, and 1,2,3,4,5-pentamethylcyclopentadiene results in ir spectral changes consistent with the formation of $Mo(CO)_2(\eta^5-C_5H_5)(\eta^3\text{-allyl})$ complexes. Establishing the role of such complexes in the formation of alkenes from 1,3-dienes using $(\eta^5-C_5H_5)Mo(CO)_3H$ is the object of continuing studies in this laboratory.

Acknowledgements

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 $\text{Mo}_2(\text{CO})_6(\eta^5\text{-C}_5\text{H}_5)_2 \xrightarrow{\text{Na/Hg}} 2[\text{Mo(CO)}_3(\eta^5\text{-C}_5\text{H}_5)]^- \xrightarrow{\text{H}^+} 2(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3(\text{H})$;
 ref. 8.
12. The $\text{Mo(CO)}_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$ isolated from the photoreaction was characterized by ir, pmr, and mass spectroscopy.
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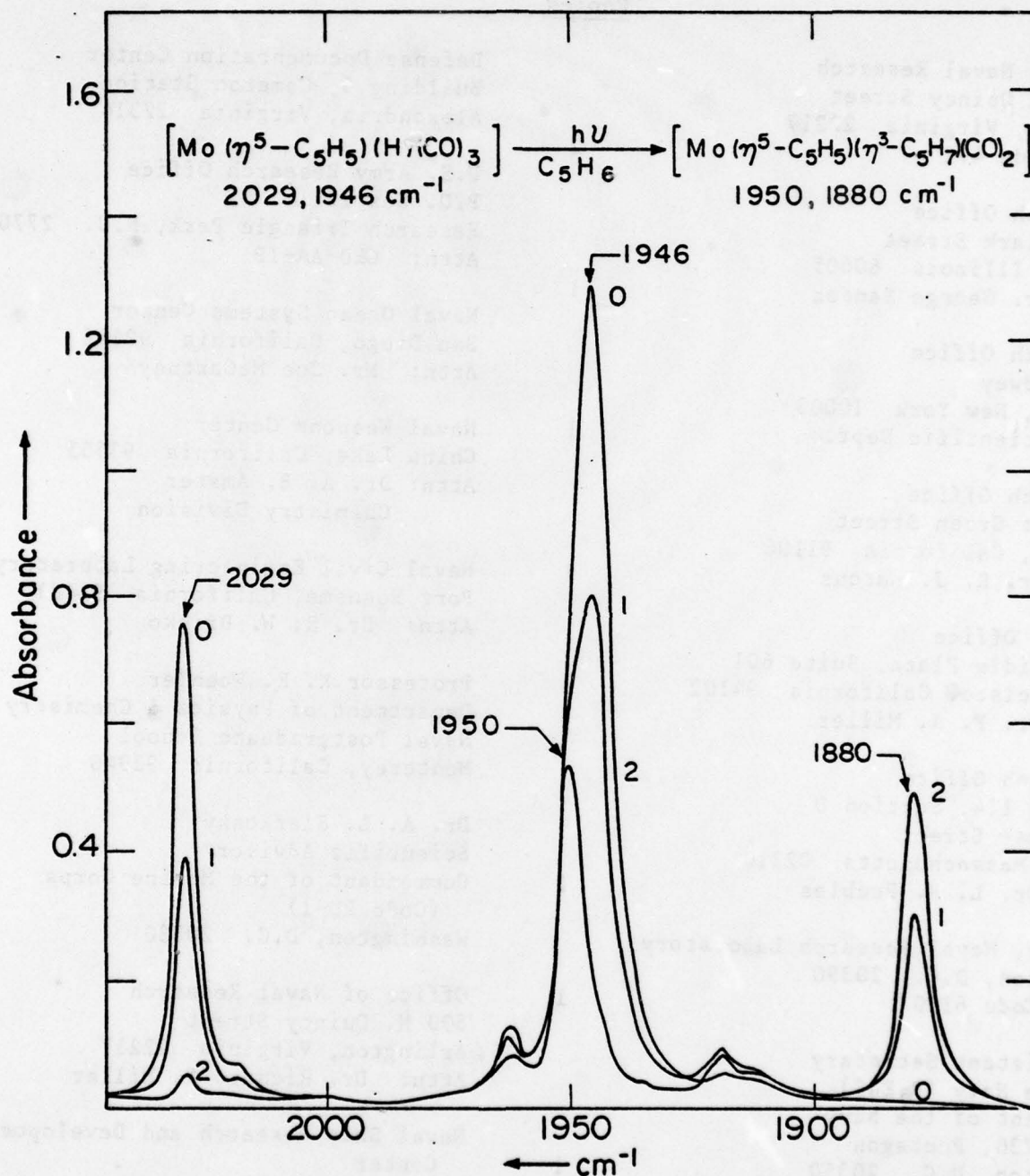
Table I. Infrared Spectral Band Maxima for Pertinent Complexes.

Complex	Band Maxima, cm^{-1} (ϵ , $\text{M}^{-1}\text{cm}^{-1}$) ^a
$\text{Mo}(\text{CO})_6$	1988 (53,800)
$(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3\text{H}$	2029 (5340); 1946 (9190)
$\text{Mo}(\text{CO})_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$	1950 (6290); 1880 (6010)
$\text{Mo}(\text{CO})_5(\eta^2\text{-C}_5\text{H}_6)$	2079 (<u>1</u>); 1957 (<u>8</u>); 1944 (<u>4</u>)
<u>trans</u> - $\text{Mo}(\text{CO})_4(\eta^2\text{-C}_5\text{H}_5)_2$	1961 (---)
$\text{Cr}(\text{CO})_6$	1987 (51,700)
$\text{Cr}(\text{CO})_2(\eta^5\text{-C}_5\text{H}_5)(\eta^3\text{-C}_5\text{H}_7)$	1942 (<u>1.14</u>); 1880 (<u>1.0</u>)

^aAll data recorded at 25°C in alkane solvent employing a Perkin Elmer 180 ir spectrometer. Numbers in parentheses which are underlined represent relative absorbance for that complex.

Figure Caption

Figure 1. Infrared spectral changes accompanying 355 ± 20 nm irradiation (2×10^{-6} ein/sec) of degassed 1.0 ml isooctane solutions of $1.4 \times 10^{-2} \text{ M}$ $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_3\text{H}$ and 0.6 M C_5H_6 . Trace 0 was before exposure to the irradiation and curves 1 and 2 are after 0.5 and 4.0 h of irradiation, respectively. Ir spectra were recorded in 0.1 mm pathlength NaCl cells.



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